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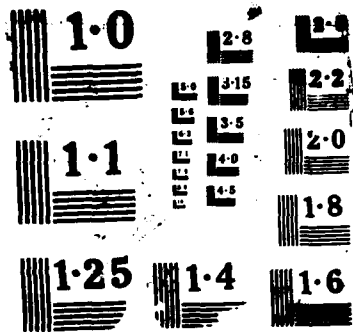
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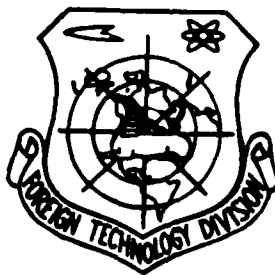
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Н н	Н н	N, n	Э э	Э э	E, e
О о	О о	O, o	Ю ю	Ю ю	Yu, yu
П п	П п	P, p	Я я	Я я	Ya, ya

*ye initially, after vowels, and after ъ, ѣ; e elsewhere.
When written as ѣ in Russian, transliterate as yѣ or ѣ.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	sinh ⁻¹
cos	cos	ch	cosh	arc ch	cosh ⁻¹
tg	tan	th	tanh	arc th	tanh ⁻¹
ctg	cot	cth	coth	arc cth	coth ⁻¹
sec	sec	sch	sech	arc sch	sech ⁻¹
cosec	csc	csch	csch	arc csch	csch ⁻¹

Russian English


rot curl
lg log


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EFFECT OF INORGANIC COMPLEXING AGENTS ON THE POLYMERIZATION OF METHYL METHACRYLATE

V. P. Zubov, M. B. Lachinov, L. I. Valuyev, V. B. Golubev,
V. A. Kabanov, V. A. Kargin

 We know that salts of metals with variable valency ordinarily inhibit radical polymerization. However, when salts such as LiCl , MgCl_2 , ZnCl_2 and AlCl_3 are present, the polymerization rate of vinyl monomers with functional groups that form coordinated complexes with salt cations rises [1-6]. The acceleration mechanism remains unknown.

The purpose of this work is to study the mechanism of polymerization in the presence of inorganic complexing agents using the polymerization of methyl methacrylate (MMA) as an example. 

The addition of AlCl_3 to MMA accelerates the polymerization of this monomer, initiated by UV irradiation in the presence of photosensitizers (benzoyl peroxide, azoisobutyric acid dinitrile (DAK)) (Fig. 1a). The polymerization rate increases up to molar ratios of

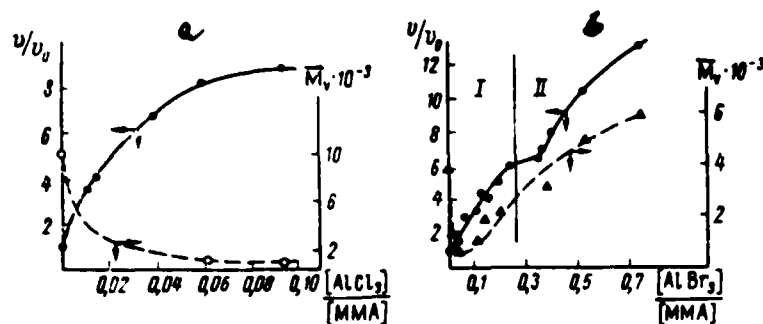


Fig. 1. Ratio of polymerization rates of MMA (v/v_0) in the presence of AlX_3 to polymerization rate of pure MMA and molecular weights of polymers as a function of the ratio $[AlX_3]/[MMA]$:

a - for $X = Cl$, 15° , $\lambda = 365$ nm, $[PB] = 3.7 \cdot 10^{-3}$ moles/l; b - for $X = Br$, 20° , $\lambda = 313$ nm.

$AlCl_3:MMA=0.1$, and then reaches the limit. The increase in the rate is accompanied by a marked reduction in molecular weight¹. Acceleration is also observed in the system $MMA-AlBr_3$ both with and without photosensitizers. The latter system is more convenient to study, since $AlBr_3$ is much more soluble in MMA. The findings are shown in Fig. 1b. At relatively small $AlBr_3$ contents, the observed dependences qualitatively agree with those shown in Fig. 1a (region I). In this region, the reaction order with respect to the intensity of the initiating radiation is 0.5, which agrees with the published data [4, 6] about the kinetics of the polymerization of MMA in the presence of $ZnCl_2$. However, a further increase in the $AlBr_3$ content causes an additional increase in the reaction rate. The molecular weight also increases and becomes even higher than during polymerization without complexing agents (region II). At the same time, we see an increase in the reaction order with respect to intensity; it reaches 0.8 with a molecular ratio of $AlBr_3:MMA = 0.7$. These facts indicate that the

¹ These data were obtained by V. V. Mal'tsev.

mechanism of the process changes during the transition from region I to region II.

It is easy to explain the abrupt increase in the polymerization rate and its approach to the maximum value at monomer:complexing agent ratios that are considerably lower than the stoichiometric ones (Fig. 1a and b, region I) if we assume that the rate constant (k_p') of the addition of the monomer to a radical bound to a complexing agent is higher than the rate constant of chain growth with the participation of a radical not bound in a complex (k_p), the breaking rate constants are the same for both radicals, and the initiation constant does not vary significantly [6]. This assumption is also supported by the fact that the photochemical decomposition rate constants of azoisobutyric acid dinitrile (DAK) in pure methyl propionate - the hydrated analog of MMA - and in a solution of $AlBr_3$ in methyl propionate (0.98 moles/l) are essentially the same, while the polymerization rate of MMA in the presence of DAK increases in the same manner as when there is no sensitizer (Fig. 1b). The examination of the corresponding kinetic picture for the range of complexing agent concentrations that are considerably lower than the monomer concentrations leads to the following expression:

$$\frac{v_0}{v - v_0} = \frac{k_p M}{(k_p' - k_p) K} \frac{1}{C} + \frac{k_p}{k_p' - k_p} \quad (1)$$

where v and v_0 are the polymerization rates with and without the complexing agent, respectively; M is the monomer concentration; C is the complexing agent concentration; K is the equilibrium constant

$R_C' + M \rightleftharpoons R' + M_C$ (where R_C' and M_C , R' and M are the radicals and monomers, bound and not bound with the complexing agent, respectively). Figure 2 shows the experimental dependences in the coordinates of equation (1). The values of K and k_p'/k_p were estimated from these dependences. For the system MMA- $AlCl_3$, $K \approx 27$, $k_p'/k_p \approx 13$; for the system MMA- $AlBr_3$, $K \approx 1.2$, $k_p'/k_p \approx 32$.

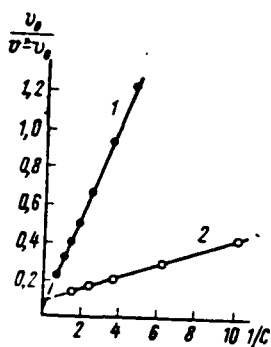


Fig. 2.



Fig. 3.

Fig. 2. $v_0/(v-v_0)$ as a function of $1/C$ for the system MMA- $AlBr_3$ (1) and the system MMA- $AlCl_3$ (2).

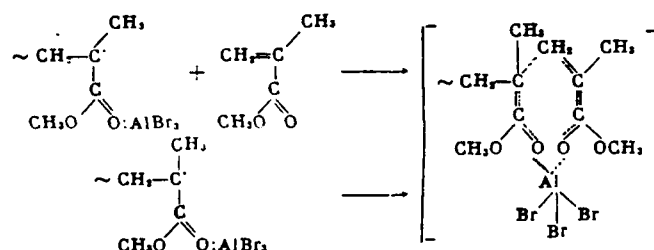
Fig. 3. EPR spectrum of trapped PMMA radicals during polymerization of pure MMA (1) and in the system MMA- $AlBr_3$ (2) with $[AlBr_3]/[MMA] = 0.9$.

In the investigation just made, we disregarded the possible change in the reactivity of the binary bonds of the monomer interacting with the complexing agent. The validity of this assumption is confirmed by the IR spectroscopic data to a certain extent. We see a considerable shift in the valent oscillation band of $C=O$ in the spectrum of the complex (1574 cm^{-1} versus 1725 cm^{-1} for pure MMA). The frequency of the valent oscillations of the binary bond varies very little (1636 versus 1645 cm^{-1} for pure MMA).

The variation in the properties of the PMMA radicals when they react with complexing agents is also confirmed by the EPR [electron paramagnetic resonance] data (Fig. 3). In the spectrum of captured radicals in the complex MMA-AlBr₃, the spectral resolution is much worse, and the width of the central line is 9 Oe greater than in the spectrum of ordinary PMMA radicals. When Al compounds whose complex formation capacity is greatly reduced (Al(iso-OC₃H₇)₃, AlBr₃·quinoline) are present, the spread of the central line does not exceed 0.8 Oe, which agrees with the theoretical estimate of the possible dipole-dipole spreading on an aluminum atom. Therefore, the changes in the EPR spectrum of radicals bound to AlBr₃ occurs because the probability of an unpaired electron staying on an aluminum atom (the magnetic moment $\mu_{Al} = 3.6409$, nuclear spin of $5/2$) is not zero. The estimate of the spin density on the aluminum atom, carried out according to the data of report [7], gives us a value of ~1%. Spreading of the central line is also observed in the systems MMA-AlCl₂ (3.3 Oe when [AlCl₃]:[MMA] = 0.8), MMA-BeBr₂ and MMA-BeCl₂ (1.6 and 0.3 Oe, respectively, when [BeX₂]:[MMA] = 1) ($\mu_{Be} = 1.1774$, nuclear spin of $3/2$). As would be expected, spreading of the lines is not observed in the spectrum of PMMA radicals obtained in the presence of ZnCl₂ (the main zinc isotopes do not have a magnetic moment). Spreading only appears when Zn⁶⁷Cl₂ is used, although the effect is small in this case because of the low value of the magnetic moment of the Zn⁶⁷ nucleus ($\mu_{Zn} = 0.8738$, nuclear spin of $5/2$). Thus, the experimental data completely agree with the assumptions about the acceleration of the chain growth reaction rate due to the reaction of the growing radicals with the complexing agent molecules.

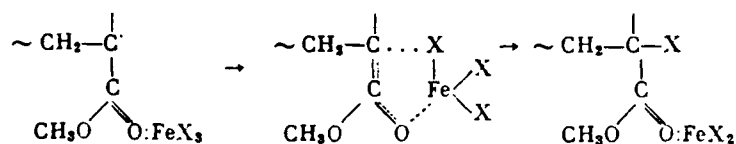
The decrease in the molecular weight of the polymer in the range of small values of the molar ratios $\text{AlBr}_3\text{:MMA}$ is obviously connected with chain transfer, in which the complexing agent molecules participate.

The mechanism of chain growth with the complexing agent can be hypothetically represented by the following diagram:



According to this diagram, the transfer of an electron to an adjoining molecule of the monomer occurs in a cyclic activated complex through the metal atom of the complexing agent. It is assumed that this event occurs more efficiently than the addition of the monomer to the radical by the usual mechanism. The transfer of an electron through a metal atom is possible for metals with unstable lowest valent states (Zn, Be, Mg, Al). For example, we know that Mg^+ is a strong electron donor [8]. The great spreading of the line or, similarly, the high unpaired electron density on the Al atom in AlBr_3 compared to AlCl_3 correlates well with the great increase in k_p with AlBr_3 established above. Naturally, in this investigation the term "radical activation" should not be interpreted in terms of the theory of ideal radical reactivity.

If the lowest valent state of the metal is stable enough, the reaction of the radical with the complexing agent molecule causes the chain to break, since the electron "is stuck" on the metal atom. This mechanism obviously occurs for salts of metals with variable valency (FeX_3 , CuX_2), e.g.:



However, the assumption of the activation of complexly bound radicals is enough to explain the acceleration of polymerization and the increase in the molecular weight and reaction order with respect to the UV light intensity at high AlBr_3 contents (Fig. 2, region II). We can assume that at high AlBr_3 concentrations, the monomer complexes form mobile ordered aggregates (labile intermediate products) [9] in which the optimum conditions for polymer chain growth are created, similarly to how this occurs in monomer liquid crystals [10] during phase transitions in solid monomers [11], etc.

Conclusions

1. The acceleration of the photoinitiated polymerization of methyl methacrylate in the presence of AlCl_3 and AlBr_3 was detected.
2. The kinetic dependences observed at small AlX_3 contents are explained by the assumption of the activation of chain growth radicals by complexing agents.

3. The properties of complexly bound radicals were investigated by EPR.

4. A mechanism of the activation of radicals by metals with unstable low valent states (ZnX_2 , AlX_3) and the inhibition of radical polymerization by metals with stable lowest valent states (FeX_3 , CuX_2) was proposed.

5. The laws governing polymerization at complexing agent concentrations that are nearly stoichiometric are explained by the origination of labile semifinished products in clusters of complexly bound monomer molecules.

Moscow State University im. M. V. Lomonosov

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STUDY OF THE COMPATIBILITY OF ACRYL SERIES OLIGOMERS
WITH POLYMETHYL METHACRYLATE

T. I. Radbil', I. N. Razinskaya, B. P. Shtarkman

The compatibility of polymers is usually determined by studying their behavior in solutions. For example, by observing the stratification of the system obtained by mixing solutions of two polymers in a common solvent one can judge the compatibility of these polymers [1]. However, this method is imperfect, since the effect of stratification will greatly depend on the concentration of mixed solutions. The method of estimating compatibility from the value of the deviation of the mixture property-composition curves on additivity has become very popular. In particular, one can study the viscosity of a solution of two polymers in a common solvent [2]. However, the opinion has been voiced [3] that the estimation of compatibility from the dependence of specific viscosity on composition may not provide an accurate representation of the actual compatibility of the system.

In this report we attempted to prove this hypothesis for amorphous polymers using polymethyl methacrylate (PMMA) - acryl series telomer systems as an example. These systems make it possible to vary the nature of the intermolecular reaction in sufficient detail by varying the molecular weight, the nature of the end groups and the value of the alkyl radicals. In this case, the compatibility of the systems was studied by several independent methods.

The selected objects of the investigation were suspended PMMA with a molecular weight of $1.65 \cdot 10^5$ (the molecular weight was determined viscosimetrically at 25° in chloroform, $[\eta] = 4.8 \cdot 10^{-5} \text{ M}^{0.8}$) and telomers of the acryl series obtained by free-radical polymerization¹. The table gives the characteristics of the telomers.

Table. Description of acryl series telomers.

(1) Исходный мономер	(2) Агент переноса цепи	(3) Мол. вес теломера	(4) Вязкость при 25°, пуазы
(5) Метилметакрилат (ММА)	(6) Изопропиловый спирт	616	—
(7) Этилакрилат (ЭА)	То же (8)	950	—
	"	1400	914
	"	2450	6700
(7) Этилакрилат (ЭА)	Изопропилбензол (9)	1600	970
	"	2150	—
(10) Бутилакрилат (БА)	(6) Изопропиловый спирт	2400	615
(11) Этилгексилакрилат (ЭГА)	То же (8)	2200	—

Henceforth the telomers obtained will be designated by adding the letter t to the abbreviation for the monomer (e.g., t. MMA, etc.).

KEY: (1) Original monomer. (2) Chain transfer agent. (3) Molecular weight of telomer. (4) Viscosity at 20°C , poise. (5) Methyl methacrylate (MMA). (6) Isopropyl alcohol. (7) Ethyl acrylate (EA). (8) Ditto. (9) Isopropylbenzene. (10) Butyl acrylate (BA). (11) Ethylhexyl acrylate (EGA).

¹ The investigated telomers were obtained by Likhterov et al.

When studying 10% solutions of ternary PMMA-telomer-chloroform systems of all the telomers given in the table, phase separation was only detected in three systems: PMMA - telomer BA [butyl acrylate]; PMMA - telomer EGA [ethylhexyl acrylate]; and PMMA - telomer EA [ethyl acrylate] with a molecular weight of 2150 obtained in an isopropylbenzene medium. The remaining systems did not exhibit significant stratification after holding for two months. At the same time, we studied the dependence of the specific viscosity of 4% solutions of PMMA and telomers in chloroform on the mixture composition. The viscosity was measured on a modified Ubellod viscosimeter with a suspended level and sealed filter at $25 \pm 0.1^\circ$.

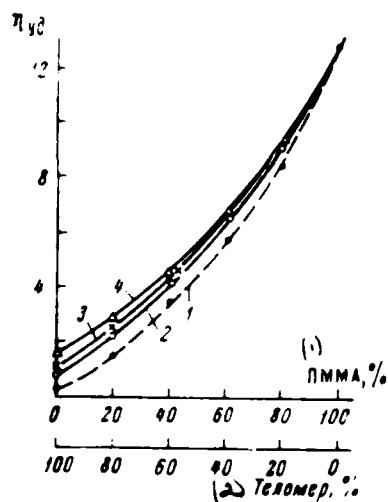


Fig. 1. Dependence of η_{sp} on composition of 4% solutions in chloroform of mixtures of PMMA with telomers:

1 - MMA; 2 - EA (mol. weight of 2450); 3 - BA; 4 - EGA.

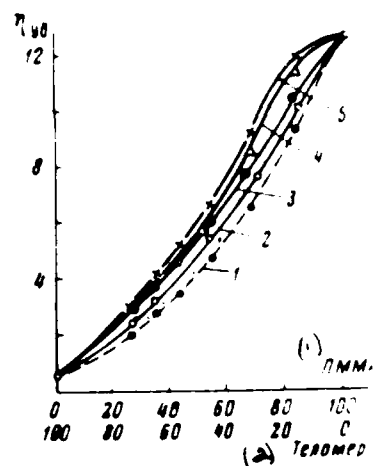


Fig. 2. Dependence of η_{sp} on composition of 4% solutions in chloroform of mixtures of PMMA and EA telomers with different molecular weights:

1 - 950; 2 - 1400; 3 - 2400; 4 - 1600; 5 - 2150 (X).

Figure 1 gives the specific viscosity - composition curves for solutions of PMMA and a series of telomers. The broken curve was

plotted by the geometric addition of the specific viscosities of a solution of PMMA and a solution of telomer MMA in chloroform [3]. The specific viscosities of the solutions of all the telomers differ little from each other, and on the scale of Fig. 1, they virtually coincide. All the curves are convex toward the abscissa and as the size of the alkyl radical in the telomer molecule increases, they are located further from the geometric addition curve; this obviously corresponds to the degradation of their compatibility with PMMA. The monotonic nature of the curves makes it possible to conclude that the process of the combination of PMMA with the telomers in question is not accompanied by the origination of specific reactions between them.

Figure 2 shows the curves of the dependence of the specific viscosities of the systems PMMA - ethyl acrylate telomers with different molecular weights and different end groups on the mixture composition. The broken curve was obtained by the geometric addition of the specific viscosities of a solution of PMMA and a solution of telomer EA with a molecular weight of 950. As one can see, the molecular weight affects the compatibility of the telomer EA with PMMA; as the molecular weight of the telomer increases, the viscosity-composition curves are deflected further and further from the additive curve. Attention is drawn to curves 4 and 5, which have a clearly pronounced S shape. The S shape of these curves is usually connected [3] with the formation of associates in the solution. The presence of isopropyl benzene end groups in the telomer EA probably makes them less compatible with PMMA because of steric factors. The limited

compatibility of PMMA and the telomers BA, EGA and EA (molecular weight of 2150) was also determined from the appearance of cloudiness in films poured from 3% solutions in chloroform on glass. Cloudiness appeared in the films with the telomers BA and EGA at a content of 5 and 1% of the telomer, respectively. The films with the other telomers remained transparent up to 40% of the telomer. We photographed the UV absorption spectra of films made from mixtures of PMMA and telomers that were dried in air and evacuated in a vacuum. The spectra were recorded on spectrophotometer SF-4. The films were 0.037 mm thick. Figure 3 shows the curves of the dependence of the transmission of certain films with a 10% content of the telomer on wavelength. The equilibrium of the PMMA - telomer system was determined from the change in the transmission of the films depending on their preliminary heating time at 60°. Figure 3b shows these curves for two telomers: EA (molecular weight of 950) and EGA. Their transmission was determined at a wavelength of 260 nm. As Fig. 3b shows, the compatibility of the system PMMA - telomer EA proved to be in equilibrium, while in the incompatible system PMMA - telomer EGA, transmission abruptly decreases as it is heated, i.e., the system is not in equilibrium.

We tried to evaluate the intermolecular interaction of the mixtures PMMA - telomer MMA, PMMA - telomer EGA in diluted solutions (0.4 g/100 ml) in a common solvent - chloroform. The characteristic viscosity $[\eta]$ and the Huggins constant (k') of samples of PMMA, telomers and different mixtures of them in chloroform were measured. Figure 4 gives curves characterizing the variation in k' with composition for the system PMMA - telomer. The broken curves were

calculated [4]. The positive deviations of the observed values of k' from the calculated values in the systems we studied indicate that there is an interaction (affinity) between PMMA and acryl series telomers in solution. Here the interaction between PMMA and the telomer EA is more intense than for the system PMMA - telomer EGA, which agrees with the data given earlier about the compatibility of the system PMMA - telomer in concentrated solutions.

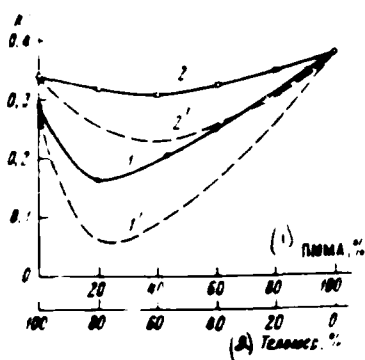


Fig. 4. Dependence of Huggins constant (k') on composition of mixtures of PMMA with telomers: EA (1, 1') and EGA (2, 2'): 1, 2 - experimental; 1', 2' - calculated data.

Thus, for amorphous polymers we see a good correlation between the deviation of the viscosity-composition dependence on additivity and the actual compatibility of the system. Therefore, the study of the dependence of the specific viscosity on composition gives us a valid idea of compatibility and makes it possible to sufficiently distinctly differentiate the characteristics of the molecular interaction in the system.

Conclusions

1. The validity of estimating the compatibility of systems of amorphous polymers by studying the dependence of the specific viscosity of the solution on composition was demonstrated using polymethyl methacrylate - telomer systems as an example.

2. The change in the nature of the intermolecular interactions of the systems in question as a result of the change in the molecular weight of the telomers, the values of their alkyl radicals and the nature of the end groups was used to show the effect of these factors on the system's compatibility.

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